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Rutgers University hosted a one-day French-American technical meeting on advanced structural ceramics on Dec. 14, 1989. Participants included seven French researchers and eight U.S. researchers from government, academia, and industry. Each participant discussed his home research institution from several viewpoints: research themes, research capabilities, and technology transfer.

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FRENCH-AMERICAN STRUCTUAL CERAMICS ROUNDTABLE - December 14, 1989

By Professor Jack Wenzel

January 23, 1991

Abstract Rutgers University hosted a one-day French-American technical meeting on advanced structural ceramics on December 14, 1989. Participants included seven French researchers and eight U.S. researchers from government, academia, and industry. Each participant discussed his home research institution from several viewpoints: research themes, research capabilities, and technology transfer.

Objective: To hold a roundtable discussion on "advanced structural ceramics" between U.S. and French researchers from government, academia, and industry for the purpose of exchanging information on the research capabilities and themes of the two countries.

Summary of meeting: **what, where, when.**

After preliminary discussions with the AFOSR (Dr. Lisa Schioler) and the French Embassy (Mr. Michel Badia) to establish an invitation list and agenda, the Institute for Engineered Materials of Rutgers University hosted a one-day technical meeting on December 14, 1989 at the new Center for Ceramic Research Building. The participants included seven French researchers and eight U.S. researchers from government, academia, and industry.

The final program is shown in Attachment A.

The meeting was preceded by an optional dinner the night before the meeting at which participants could meet each other for the first time in an informal setting.

Participant List

The list of participants follows. Copies of business cards giving business affiliations and addresses may be found in Attachment B.

FRENCH

M. Badia	Attache Scientifique, Ambassade de France
C. Bonami	Direction des Recherches, Etudes et Techniques d'Armement (D.R.E.T.)
J.L. Chermant	LERMAT-ESMRA-Universite de Caen

Y. Honnorat	SNECMA, Chef du Departement "Materiaux et Procedes"
P. Lamicq	Societe Europeenne de Propulsion (S.E.P.), Directeur Scientifique
P. Merle	Groupe d'Etudes Metallurgie Physique et Physique des Materiaux - INSA LYON
P. Poirier	Responsable du Developpement long terme du Departement CERAMIQUES de RHONE-POULENC CHIMIE

U.S.A.

James A. Cornie	Director, Metal Matrix Composites Laboratory Massachusetts Institute of Technology
Charryl Greenwood	Contracts/Licensing Manager, Industrial Applications Office, Los Alamos National Lab
D. Ray Johnson	Manager, Ceramic Technology Project Oak Ridge National Laboratory
Malcolm G. McLaren	Rutgers University, Institute for Engineered Materials
Marc Newkirk	President, Lanxide Corp.
Maxine L. Savitz	Director, Garrett Ceramic Components
Lisa Schioler	Air Force Office of Scientific Research
Jack Wenzel	Rutgers University, Center for Ceramic Research

Summary of Presentations

The U.S. participants discussed their home institutions from three points of view: research themes in the area of structural ceramics; research capabilities, especially equipment for characterization and processing; and (in the case of the universities and national laboratories) technology transfer practice. In addition, the Lanxide representative spoke about industrial interactions with governnment and university, and about government policies which could help or hinder the development and commercialization of advanced ceramics in the United States.

The French participants discussed their home institutions from a slightly different point of view: research themes in the area of structural ceramics, and research capabilities. Little mention was made of technology transfer practices from government or academic laboratories to industry. One participant (Jean-Louis Chermant from the University of Caen) gave a more comprehensive presentation in which he reviewed research in all the major French academic laboratories involved in using ceramic fiber ceramic matrix composites. A copy of a paper on which this presentation was based may be found in Attachment C.

Publications

Although there were no formal proceedings published of this one-day meeting, most particpants distributed previous publications concerning their laboratories and their research. One such publication is cited in Attachment C.

Recommendations for future meetings

The participants were of the unanimous opinion that the roundtable was a success in allowing researchers on both sides of the Atlantic to become better acquainted with each others research programs in structural ceramics. The seeds of long-range collaborations have been sown. The U.S. participants thought the meeting worthwhile and were pleasantly surprised at the advanced stage of French research in structural ceramis. It is hoped that this meeting has thereby furthered U.S. and AFOSR research goals.

ATTACHMENT A

U.S. French Advanced Ceramics Roundtable
on
Industry-Government-University Cooperation and Technology Transfer
December 14, 1989

Center for Ceramic Research
Rutgers University
Piscataway, NJ 08855

REVISED PROGRAM

8:00	Coffee
8:15	Welcome - Malcolm G. McLaren, Rutgers University - Michel Badia, French Embassy - Lisa Schoiler, AFOSR
8:30	J. L. Chermant, University of Caen
9:00	J. Wenzel, Rutgers University
9:20	P. Merle, INSA Lyon
9:40	J. Cornie, MIT
10:00	Coffee
10:30	P. Poirier, Rhone-Poulenc
10:50	M. Newkirk, Lanxide
11:10	P. Lamicq, SEP
11:30	M. Savitz, Garrett
12:00	Lunch
1:00	C. Bonami, DRET
1:20	R. Johnson, ORNL
1:40	Y. Honnorat, SNECMA
2:00	C. Greenwood, LANL
2:20	Coffee
2:50	Open Discussion - Moderator: J. Wenzel
3:30	Summary and Conclusions - L. Schoiler and M. Badia
4:00	End of Meeting

ATTACHMENT B

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ATTACHMENT C

JAP-CMC-89

RESEARCH ON CERAMIC MATRIX COMPOSITES IN FRANCE

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ABSTRACT

This paper gives an outline of the research activity of the main French academic laboratories and companies involved with long ceramic fibers in ceramic matrix materials. Some aspects of processes, microstructure, morphology and mechanical properties and behavior are described.

1. INTRODUCTION

Parts for aeronautics and space applications require materials withstanding very high temperatures, higher than 1500°C. This range of temperatures corresponds to a domain where neither classical superalloys (monocrystalline, such as for the M 88 motor of the Rafale aircraft, or otherwise) nor even monolithic ceramics can work (Fig. 1), (1) (2). Moreover, for such parts, structural weight savings are directly translated into increases in payload, into fuel economy and efficiency, and into the range of travel distance. Then one possibility is to coat these materials to support higher temperatures, but most often they can only be used for a short term period. One can also develop ceramic composite materials which are tougher than monolithic ceramics, i.e. less brittle, but these will have to be coated for applications at the highest temperatures.

They consist of laminated structures, made of layers of ceramic fiber cloth, infiltrated or embedded in a carbon or ceramic matrix, and which can receive a ceramic coating. In these conditions, the fibers provide high strength and some stability at high temperature, the matrix brings the rigidity and the coating provides the resistance to oxidation and to atmospheric environment. These materials are stronger than steel, stiffer than titanium and lighter than aluminum or aluminum-lithium alloys. Resort to such materials is common place when resistance to heat and ablation is critical (4).

Through the impetus given by the Société Européenne de Propulsion (SEP) and the Aérospatiale Companies, France takes presently the position of leadership to produce industrially CMC materials, from carbon fibers produced essentially in Japan or in the USA, or from silicon carbide fibers produced in Japan. Some alumina or mullite CMC materials are also developed. Recently the Rhône-Poulenc Company developed a new fiber of silicon carbo-nitride, which looks very promising.

From 1984 to 1988, a French Program Contract on the mechanical behavior of CMC materials was supported by the National Center of Scientific Research (Centre National de la Recherche Scientifique, CNRS), the Direction of Research, Investigations and Technics (Direction des Recherches, Etudes et Techniques, DRET) and the Materials Department of the Research and Higher Education Department (Département Matériaux du Ministère de la Recherche et de l'Enseignement Supérieur, MRES), with the participation of two companies : Aérospatiale and SEP, and six laboratories : EMP - Corbeil, GEMPPM - Villeurbanne, ENSCI - Limoges, LCS - Bordeaux, LERMAT - Caen, ONERA - Chatillon sous Bagneux. Presently two Scientific Groups of CNRS (Groupement Scientifique, GS) with SEP and with Aérospatiale and SEP Companies are supporting these researches, together with DRET and MRES.

The ever-growing interest on CMC is also reflected in the number of specific meetings or collecting publications, which focus specifically on these advanced ceramic materials (5) (6) (7) (8) (9).

The aim of this paper is a brief outline of the French research activity of academic laboratories and companies, focussing essentially on process routes, microstructure and morphology, as well as mechanical behavior of CMC materials. From the documents given by these different laboratories, I shall try to make a presentation of the various tasks performed successively in main industrial and academic laboratories, with an emphasis on their main results.

Fig. 3 : Nose in multidirectionnal carbon with a protection for the HERMES shuttle (with the authorization of Aérospatiale).

2.2 CMC composites

As C-C composite materials present also some other limitations in their use (physical properties insufficient, too sensitive to the environment), to solve this key problem, ceramic matrix composite materials have been developed and are produced, more specifically under the impetus of SEP Company. They are, for example, C-SiC (SEPCARB[R] INOX) and SiC-SiC (CERASEP[R]) composites, first developed by SEP nearly 10 years ago. Following a development and qualification testing period, these materials will soon enter the series production phase. They can be considered complementary to the SEPCARB[R] product line, since they offset these two limits through their superior physical properties (higher shear, compression and tensile strength, ...) as well as being far less sensitive to oxidation. Some characteristics of these materials are :

- SEPCARBINOX[R] (C-SiC) can be used for long periods at temperatures up to 1600°C-1700°C ; for example, after 15 tests at 1550°C followed by 2 tests at 1750°C under air in the Almeria Solar Furnace, the weight less is only less than 2% and the mechanical characteristics and stiffness remain more than 80% ; these materials possess the best physical properties, and protective treatments exist for this maximum temperature range,
- CERASEP[R] (SiC-SiC) can be used for very long periods at temperatures up to 1200°C-1300°C ; they are less sensitive to oxidation.

* For short or medium - length periods, we can add from 200° to 500°C to the operational temperatures indicated above.

- class 1000 (°C) with carbon and SiC reinforced glass-ceramic,
- class 1500 (°C) with SiC whiskers and carbon fibers reinforced ceramic (SiAlYON, silicon nitride and carbide, silica).

These developments are based on the latest process including performances, parts, cost and time aspects. SiC whiskers reinforced SiAlYON and Si_3N_4 are presently obtained by HP or HIP process. They are developed specially for spacial parts and high temperature joining. Before the end of the year, Aérospatiale Company with Céramiques & Composites (Sub-company of Aérospatiale and Rhône-Poulenc Companies) is planning to present two new industrial composites (1 of each class).

Aérospatiale Company has also developed 3D Nextel-SiO_2 composites (radioelectric applications) and other high temperature, high performance CMC structures.

Very well-known as a leading European Company in the field of advanced ceramics (Al_2O_3, ZrO_2 (PROZYR[R]), Si_3N_4 KERSIT trade mark), Céramiques Techniques Desmarquest Company is also developing CMC materials based on SiC whiskers and on chopped or long ceramic fibers. These include :

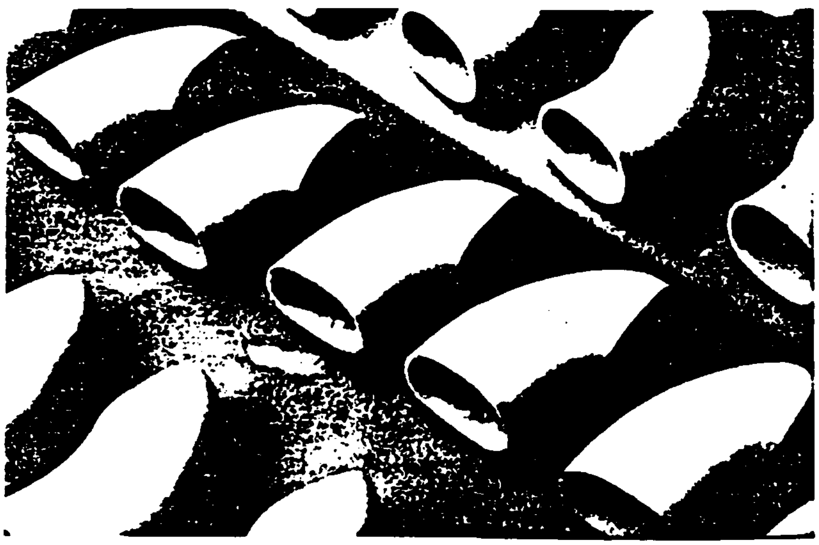

Fig. 5 : Exhaust pipes in silica reinforced by chopped SiC fibers (with the authorization of Céramiques Techniques Desmarquest).

- fibrous ceramic structures : their production method allows to manufacture structural components with various combinations of pure silica (99,99%) and pure alumina (96%) fibers ; the fibrous structure is reinforced by a specific inorganic binder ; fiber volume fraction can be tailored to meet special require-

LCTS is involved in research on processing according to both the gas phase route (e.g. chemical vapor infiltration process, CVI) and liquid phase route (organic precursor/ceramic transition).

As far as the gas phase route is concerned, this laboratory has worked out the isothermal/isobaric CVI process used to make CMC from a porous fiber preform and a gaseous precursor of the ceramic matrix. The ceramic matrix is deposited in the pore network of the preform as the result of a chemical reaction involving gaseous species. In order to favor in-depth deposition with respect to surface coating, deposition has to be performed at low temperature and pressure. It is a rather slow process, but a large number of preforms (even of different and complex shapes) can be treated simultaneously. The process has been successfully applied to carbides (SiC, B_4C, TiC), nitrides (BN) and oxides (Al_2O_3, ZrO_2). It has been transferred to industry (SEP, Du Pont De Nemours) for silicon carbide.

In the field of the liquid phase route (another promising processing route) the research is directed towards a fundamental study of the transition that occurs, under the effect of temperature, between polycarbosilane (or polycarbosilazane) precursors and the ceramic state. The transition is studied on the basis of TGA, gas analysis, IR, RAMAN, ESCA, XRD, EXAFS, TEM analyses as well as electrical measurements. Finally, the effect of the precursor chemistry, pyrolysis conditions, ceramic microstructure, on the mechanical properties of the ceramic is investigated from tensile tests performed on monofilaments.

ONERA is currently developing also several ceramic composite shaping techniques, applied to thick three-dimensional structures of ceramic and carbon fibers. Four such techniques are developed :

- i) chemical vapor infiltration (CVI),
- ii) densification of fiber yarn surrounded with ceramic glass by hot pressing,
- iii) infiltration and sintering of ultrafine powders,
- iiii) organometallic process, i.e. injection of colloidal gel for oxides and pyrolysis of polymers for covalent ceramics.

These different techniques were brought together in 1987 to develop new processes that would improve the quality of the matrices and reduce the manufacturing time. Of all these new processes, the first (CVI) is used only for the surface treatment of fibers in the fibrous preforms, to control the fiber/matrix interface, e.g. for CVI carbon.

- determination of the elastic constants of anisotropic systems by measurement of longitudinal and shear velocities in given directions ; this is generally performed at high frequency (20 MHz),
- measurement of unidirectionnal elastic modulus, E, from 20°C up to 1700°C, using longitudinal "long beam" ultrasonic modes at low frequencies (80 KHz - 350 KHz) ; this equipment can also operate under regulated pressure (0.05 to 1000 mbar) and controlled atmosphere (oxidizing or reducing).

This is of much interest because structural ceramic composites are used at high temperature : it is a non destructive test of the materials in service conditions.

While the knowledge of the elastic behavior in service conditions is important for engineers, the measurement of the variations of E with temperature is a useful tool to study structural modifications or damage, when composites are cycled or aged with temperature : phase transformations (ex. crystallization of amorphous matrix), interface alteration (oxidation), densification by sintering effects, microcracking due to thermal stresses.

Two classes of materials are investigated : a *2D SiC-SiC* composites (SEP origin) and 3D SiC or Nextel in SiO_2 matrix (Aérospatiale origin). The factors under investigation are essentially the effect of atmosphere, temperature and pressure on the fiber/matrix interface, and the structural stability domain of the matrix.

6. RUPTURE AND TOUGHNESS EVALUATION

The macroscopic fracture behavior is investigated at room temperature by several laboratories (EMP, GEMPPM, LCTS, LERMAT and ONERA) on SENB, DCB or CT specimens. Materials investigated are more specially : i) C-C, C-SiC, SiC-SiC, from SEP origin, ii) SiC-SiO_2, 3D Nextel-SiO_2, 1D SiC-mullite from Aérospatiale origin. Research has been directed at the question of whether or not linear elastic fracture mechanics (LEFM) concepts can be applied to these coarse anisotropic structures. Various fracture energy parameters (elastic, non-elastic, ...) and crack growth resistance curves are determined. Acoustic emission is also often used to inform on the damaging.

The shape of the R curve is typically due to the extending of a process zone at crack front (matrix cracking, fibre breaking and pull-out, ...) and then to the sliding of the process zone with crack extension, giving a plateau of crack resistance. For higher crack increment there is a specimen end effect which depends on the size of the process zone with regard to the specimen ligament. For

7. MICROSCOPIC MECHANICAL APPROACH

A better understanding of the CMC fracture behavior requires microscopic approaches and a discrimination between the respective roles of the various components : fibers, matrix and interface. That is underway at EMP, GEMPPM, LERMAT and ONERA.

Micromechanical interface behavior measurements are performed by micro-indentation technique (EMP, GEMPPM, LERMAT and ONERA) connected with acoustic emission, and by tension (ONERA) or bending tests (LERMAT) in situ. In the case of strong interface, it was shown that the debonding criterion is linked to energy balance and the critical fiber debond stress is depending on fiber diameter. This can be easily checked with SiC fibers because they have a large scatter in diameter. Quantitative acoustic emission gives access to the dynamics of debonding process. In the case of weak, frictional interface, indentation leads to the critical shear stress at interface. Due to thermal strain misfit, this parameter depends on the details of surroundings of the tested fiber and on the nature of the fiber coating.

The fiber pull-out mechanism depends on interface characteristics and on fiber properties (average strength, Weibull modulus). The analysis of fracture surfaces brings information about these parameters and about the stress transfer conditions around the matrix crack.

Instead of traditional fracture mechanics parameters developped for ductile materials, an original approach was preferred at EMP, taking into account the discontinuous structure of these types of materials. The impregnated yarn was supposed to be the elementary constituent for characterizing the damage and the fracture of the material. Common local behaviour- and damaging-laws allowed the experimental results to be explained for unnotched specimens as well as for notched ones.

The analysis of the energy dissipated during the fracture showed that an important part of it derived from structure distorsions in the volume of the material. Finally, the numerous heterogeneities of the structure (porosity, weaving flaws, ...) are at the origin of a great scatter in the results.

From an experimental point of view, experiments are performed on SiC-SiC composites, using extensometry and acoustic emission:

- tensile tests on impregnated yarns to determine their mechanical behaviour, damage and fracture laws analyzed using original statistical functions ; these experiments are also useful to

9. MICROSTRUCTURE AND INTERFACES

A general feature of the above results is the drastic role of the microstructure and of the interfaces. Moreover during high temperature uses or tests, a change in the chemical content of the fibers and of the interfaces arises. This evolution and the microstructure characteristics control, in fact, the strength of these materials. The main laboratory involved in this direction is the LMM in Pau, which uses analytical methods, such as high resolution transmission electron microscopy (HRTEM), X-R scattering, laser beam optical scaterring. Besides carbon fibers and precursors and carbon-carbon composites, the LMM investigates SiC and Si_3N_4 based continuous fibers and precursors, whiskers, ceramic matrix and yarn reinforced or hot-pressed composites, as received or after various thermal treatments.

Among others, recent results have been concerned with the role of the free carbon, of the oxygen and of the SiO_xC_y tetrahedra in SiC-based materials. For example, the behavior of the aromatic free carbon has been found to govern the electrical properties in a ceramised polycarbosilane. The SiO_xC_y tetrahedra have been found to act as grain boundaries hindering the SiC crystal growth in the Nicalon fiber. The change in the β SiC nanocrystals in the Nicalon fibers as a function of temperature has also been investigated. β SiC crystals have been found to decompose at low temperature (1000-1200°C), giving labile Si which is able to form SiO_2 at the fiber surface. In another domain, sequential deposits of various phases (C, SiO_2, SiC) have been observed during the vapor deposition of SiC or carbon on Nicalon fiber. The knowledge of these structural information is very important and has been correlated to the mechanical behavior.

Transmission electron investigations are also performed at LERMAT, in close connection with mechanical test results, to get information on the role of the matrix, the fibers and the interfaces and on their change during mechanical test.

At LPM the microstructure and the chemical composition of CMC are characterized by electron microscopy (TEM, HRTEM, EELS, EDX), electron probe and SIMS. SiC-SiC developed by ONERA were investigated : SIMS analyses have resulted in an efficient control of the change in the fiber composition which occurs during the process. The microstructure and the composition of the reaction layer are also characterised by HRTEM and EELS on SiC-LAS composites developed by ONERA and Saint-Gobain Recherche.

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APPENDIX

The different laboratories quoted in this paper are :

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Fig. 1 : Mechanical strength of different high temperature materials and change in the type of materials for thermomechanical applications from 1960 (SA : superalloys ; DS : directional solidification materials ; CVD : chemical vapor deposited materials ; CMC ceramic matrix composites).

Fig. 2 : Temperatures distribution on the HERMES intrados for the following conditions : flight speed : mach 29 ; altitude 79 km with an angle of 28°5 (from a CNES document).

Fig. 3 : Nose in multidirectionnal carbon with a protection for the HERMES shuttle (with the authorization of Aérospatiale).

Fig. 4 : Leading-edge in C-SiC for the HERMES shuttle (with the authorization of SEP).

Fig. 5 : Exhaust pipes in silica reinforced by chopped SiC fibers (with the authorization of Rhône-Poulenc Chimie).